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Research Article

Sorption of Pb^{2+} from Aqueous Solution unto Modified Rice Husk: Isotherms Studies

A. O. Dada,[1] J. O. Ojediran,[2] and Abiodun Paul Olalekan[3]

[1] *Department of Chemistry, Landmark University, P M B 1001, Kwara State, Omu-Aran 370102, Nigeria*
[2] *Department of Agricultural and Biosystems Engineering, Landmark University, P M B 1001, Kwara State, Omu-Aran 370102, Nigeria*
[3] *Department of Chemical Engineering, Landmark University, P M B 1001, Kwara State, Omu-Aran 370102, Nigeria*

Correspondence should be addressed to A. O. Dada; dada.oluwasogo@landmarkuniversity.edu.ng

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Investigation of the sorption potential of rice husk, an agricultural waste, as an adsorbent was carried out. The rice husk was modified with orthophosphoric acid and was used for adsorption of lead (II) ions (Pb^{2+}) from aqueous solution. Physicochemical properties of the modified rice husk were determined. Equilibrium sorption data were confirmed with Langmuir, Freundlich and Temkin adsorption isotherms. On the basis of adsorption isotherm graphs, R^2 values were determined to be 0.995, 0.916, and 0.797 for Langmuir, Temkin, and Freundlich isotherms, respectively, indicating that the data fitted well into the adsorption isotherms, but Langmuir isotherm is a better model. The maximum monolayer coverage from Langmuir studies, $Q_{max} = 138.89$ mg/g, Langmuir isotherm constant, $K_L = 0.699$ L/mg, and the separation factor, $R_L = 1.41 \times 10^{-2}$ at 100 mg/L of lead(II) ions indicating that the sorption process, was favourable. The suitability of modified rice husk as an adsorbent for the removal of lead ions from aqueous solution and its potential for pollution control is established.

1. Introduction

The pollution of water resources due to the disposal of heavy metal ions has been an increasing worldwide concern for the last few decades. It is well known that some metals are poisonous or otherwise toxic to human beings and ecological environments as reported by Abdel-Halim and coresearchers [1]. Increasing levels of heavy metals and other pollutants in the environment pose serious threats to water quality, human health, and living organisms. Lead(Pb) is considered as one of the priority metals from the point of view of potential health hazards to human, and it is listed by the Environmental Protection Agency (EPA) as one of 129 priority pollutants. The case of recent lead poisoning of hundreds of children in Zamfara State has been identified as the worst lead poisoning in Nigeria's history [2].

There are various methods of removing heavy metal ions, and they include chemical precipitation, membrane process, ion exchange, solvent extraction, electrodialysis, and reverse osmosis [3]. These methods are noneconomical and have many disadvantages such as incomplete metal removal, high reagent and energy consumption, and generation of toxic sludge or other waste products that require disposal or treatment. In contrast, the adsorption technique is one of the preferred methods for the removal of heavy metal ions because of its efficiency and low cost [4]. For this purpose, in recent years, interest has recently arisen in the investigation of some unconventional methods and low-cost materials for scavenging heavy metal ions from industrial waste waters [5] using low-cost natural adsorbents which are economically viable such as agricultural wastes. In general, an adsorbent can be assumed as "low cost" if it requires little processing, is abundant in nature, or is a by-product or waste material from industry [6]. Some of the reported low-cost adsorbents include bark, tannin-rich materials, lignin, chitin, chitosan, peat moss, moss, and modified wool and cotton. Insoluble starch xanthates have been found to be very useful to remove heavy metal ions from solutions [7]. Agricultural waste materials such as spent grain [8], polymerized onion skin [9] including sunflower stalks [10], wood cellulose [11], maize bran [3], coconut shell, waste tea, rice straw, tree leaves, peanut and walnut husks, and other adsorbents like

goethite [12] and manganese hexacyanoferate(II)/(III) [13] have been studied by various researchers to investigate their effectiveness in binding heavy metal ions. The adsorption of heavy metals by these materials might be attributed to their proteins, carbohydrates, and phenolic compounds which have carboxyl, hydroxyl, sulphate, phosphate, and amino groups that can bind metal ions.

Rice is the second largest produced cereal in the world. Rice husk is the hard protecting covering of grains of rice. It is an agricultural waste material obtained from the threshing of the rice and constitutes about 20% of 650 million tons of rice produced annually in the world [14]. Although low in calorific value, rice husk is used as fuel for some industrial and household purposes. In addition to protecting rice during the growing season, rice husk can be put to use as building material, fertilizer, insulation material, or fuel. It is used for production of mesoporous sieves which find application in catalysis, as a support for drug delivery system and as an adsorbent in waste water treatment. In fact, it was investigated as a viable material for the treatment of synthetic direct red dye in industrial waste water [15–17].

Most commonly used bioadsorbents are untreated/unmodified; however in this research, the potential of acid modified rice husk is investigated in the removal of toxic heavy metal ion such as lead from its aqueous solution namely, the study of the Langmuir, Freundlich, and Temkin adsorption isotherms.

2. Material and Method

Collection and Preparation of Adsorbent. Rice husk was obtained from a local mill in Ilorin, Kwara State and was pretreated according to the method reported by Milind et al. [18] and Ken-Sen et al. [19]. The rice husk was screened and washed with deionized water to remove dirt and metallic impurities after which it was dried in the oven at about 105°C for 2 hours. The dried rice husk was grounded and sieved in the mesh in the range between 250 μm and 150 μm in order to increase its surface area. The sieved rice husk was treated with 1.0 M orthophosphoric acid (H_3PO_4) and heated on the magnetic stirrer at 100°C until it totally formed a paste. The modified rice husk was washed with de-ionized water until the pH $\approx$ 6. It was later dried in the oven at about 80°C to remove moisture. The modified adsorbent was tagged phosphoric acid modified rice husk (PRH).

The PRH was characterized by determining the following parameters: specific surface area, moisture content, loss of mass on ignition, pH, and bulk density using standard procedures.

2.1. Simple Specific Surface Area Determination. Saers method has been used by a number of researchers [20–23]. This method is accurate in the determination of surface area of rice husk because the effect of chemical modification was characterized using this method. A sample containing 0.5 g of the PRH was acidified with 0.1 M HCl to pH 3–3.5; the volume was made up to 50 cm^3 with de-ionized water after addition of 10.0 g of NaCl. The titrations were carried out with standard 0.1 M NaOH in a thermostatic bath at 298 ± 0.5 K to pH 4.0 and then to pH 9.0. The volume V required to raise the pH from 4.0 to 9.0 was noted, and the surface area was computed from the following equation:

$$S\left(\frac{m^2}{g}\right) = 32V - 25. \quad (1)$$

2.2. Determination of the Moisture Content. 5 g of the PRH was weighed into a crucible. This was placed in the oven and heated for 5 hrs at constant temperature of 105°C. The sample was then removed and put rapidly into a desiccator in order to prevent more moisture uptake from atmosphere. The sample was reweighed. This procedure was repeated several times until a constant weight was obtained. The difference in the mass constitutes the amount of moisture content of the adsorbent [24]:

$$\%\ \text{moisture content} = \frac{w_2 - w_3}{w_2 - w_1} \times 100, \quad (2)$$

where

w_1 = weight of crucible,

w_2 = initial weight of crucible with sample,

w_3 = final weight of crucible with sample.

2.3. The Determination of Loss of Mass on Ignition. This was done by weighing 10 g of the adsorbent and put inside furnace at constant temperature of 600°C for 2 hrs. After roasting, the sample became charred and was removed from the furnace then put in a desiccator for cooling. The residual product is then weighed, and the difference in mass represented the mass of organic material present in the sample. This operation was repeated four times.

2.3.1. pH Determination. pH of the samples was determined by weighing 1 g each of PRH, boiled in a beaker containing 100 mL of distilled water for 5 min; the solution was diluted to 200 mL with distilled water and cooled at room temperature. The pH of each was measured using a pH meter (model ATPH-6), and the readings were recorded [1].

2.3.2. The Bulk Density. Archimedes' principle was used to simply determine the bulk density by weighing a 10 cm^3 measuring cylinder before and after filling with the samples. The measuring cylinder was then dried, and the sample was packed inside the measuring cylinder, leveled, and weighed. The weight of the sample packed in the measuring cylinder was determined from the difference in weight of the filled and empty measuring cylinder. The volume of water in the container was determined by taking the difference in weight of the empty- and water filled-measuring cylinder. The bulk density was determined using the equation below [25]:

$$\text{bulk density} = \frac{w_2 - w_1}{v}, \quad (3)$$

where

w_1 = weight of empty measuring cylinder,

w_2 = weight of cylinder filled with sample,

v = volume of cylinder.

The preparation of adsorbate was carried out by preparing stock solution containing 1000 mg/L of Pb. 0.3998 g of $Pb(NO_3)_2$ in 250 cm^3 of de-ionized water. Working concentration in the range of 10 mg/L–200 mg/L was prepared by serial dilution.

2.3.3. Sorption Experiment. The equilibrium sorption of the Pb^{2+} ions unto PRH was carried out by contacting 0.1 g of the substrate with 100 cm^3 of different concentrations from 10 mg/L–200 mg/L in 250 cm^3 pyrex conical flask intermittently for 90 minutes. The mixture was filtered, and the residual concentration of the filtrate was analyzed using Atomic Absorption Spectrophotometer (2380 UNICAM AAS). The amount of adsorbed (mg/g) was calculated using the formulae reported by Vanderborght and Van Grieken [26]:

$$Q = \frac{v\left(C_i - C_e\right)}{w}, \tag{4}$$

where Q = the amount of solute adsorbed from the solution, v = volume of the adsorbate, C_i = the concentration before adsorption, C_e = the concentration after adsorption, and w = the weight in gram of the adsorbent. The data were fitted into the following isotherms: Langmuir, Freundlich, and Temkin [27]. The removal efficiency was determined by computing the percentage of sorption using the formulae in (5):

$$\%\ \text{sorption} = \frac{C_i - C_e}{C_i} \times 100. \tag{5}$$

3. Results and Discussion

The physicochemical parameters of the phosphoric acid modified rice husk (PRH) are shown in Table 1.

The physicochemical parameters of the modified rice husk are, namely, pH, % moisture content, % loss of mass on ignition, bulk density (g/cm^3), particle sizes, and surface area (m^2/g). The values reported are in the range with those reported in the literature [14].

3.1. Sorption Isotherms of Pb(II) Ion unto PRH. The equilibrium sorption of the Pb^{2+} ions was carried out by contacting 0.1 g of the PRH with 100 cm^3 of 1000 mg/L of different concentrations from 10 mg/L–200 mg/L in 250 cm^3 Pyrex conical flask intermittently for 90 minutes on the orbital shaker. The mixture was filtered, and the filtrate was analyzed for metal ions concentration using AAS. The data were fitted into the following isotherms: Langmuir, Freundlich, and Temkin.

The adsorption data obtained with the adsorbent correlates well with Langmuir, Freundlich, and Temkin adsorption models and were illustrated in Figures 1, 2, and 3. The Langmuir equation was chosen for the estimation of maximum

Table 1: Some physicochemical parameters of the phosphoric acid modified rice husk (PRH).

Properties	PRH
pH	6.30–6.50
% Moisture content	12
% Loss of mass on ignition	0.9
Bulk density (g/cm^3)	0.386
Particle size	300 μm < Φ < 250 μm
Surface area (m^2/g)	198

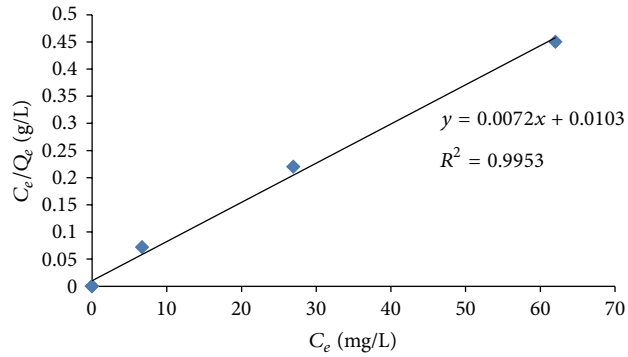

Figure 1: Langmuir adsorption isotherm of Pb(II) ion unto PRH.

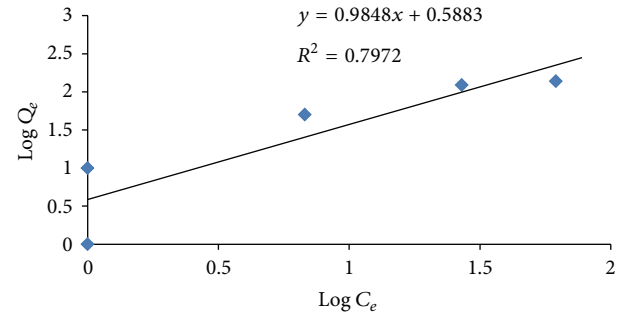

Figure 2: Freundlich adsorption isotherm of Pb(II) ion unto PRH.

adsorption capacity corresponding to complete monolayer coverage on the PRH surface [27]. The Langmuir model assumes the surface of the sorbent to be homogenous and the sorption energies to be equivalent for each sorption site. The essential characteristics of the Langmuir model can be expressed in terms of a dimensionless constant and separation factor or equilibrium parameter, R_L. From the results in Table 3, the R_L values were found to be greater than zero and less than one, that is, $0 < R_L < 1$. R_L value between 0 and 1 indicates favorable adsorption. This means that a favorable adsorption was observed in this study. Langmuir adsorption describes quantitatively the formation of a monolayer of adsorbate on the outer surface of the adsorbent, and after that, no further adsorption takes place. Thereby, the Langmuir represents the equilibrium distribution of metal ions between the solid and liquid phases [28]. The Langmuir isotherm is valid for monolayer adsorption onto a surface

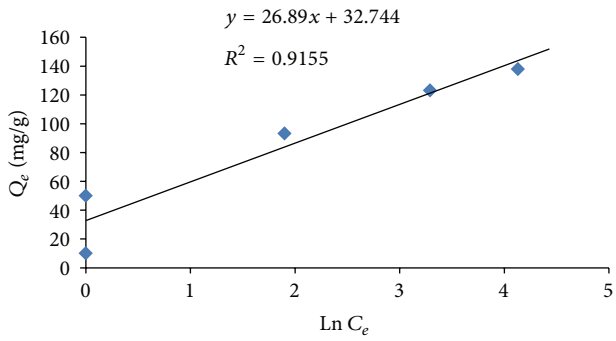

Figure 3: Temkin adsorption isotherm of Pb(II) ion unto PRH.

containing a finite number of identical sites. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. Based upon these assumptions, Langmuir represented the following equation:

$$q_e = q_{\max} \frac{K_L C_e}{1 + K_L C_e}. \quad (6)$$

Langmuir adsorption parameters were determined by transforming the Langmuir equation (6) into linear form:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}}, \quad (7)$$

where

C_e = the equilibrium concentration of adsorbate (mg/L^{-1}),

q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g),

$q_{\max}$ = Langmuir constants related to adsorption capacity (mg/g),

K_L = rate of adsorption (L/mg). The values of $q_{\max}$ and K_L were computed from the slope and intercept of the Langmuir plot of C_e/q_e versus C_e [24]. The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter R_L, which is a dimensionless constant referred to as separation factor or equilibrium parameter [29]:

$$R_L = \frac{1}{1 + (1 + K_L C_0)}, \quad (8)$$

where

C_0 = initial concentration,

K_L = the constant related to the energy of adsorption (Langmuir constant), R_L value indicates the adsorption nature to be either unfavourable if $R_L > 1$), linear if $R_L = 1$, favourable if $0 < R_L < 1$, and irreversible if $R_L = 0$. From the data calculated in Table 3, the R_L is greater than 0 but less than 1 indicating that Langmuir isotherm is favourable.

Freundlich model was chosen to estimate the adsorption intensity of the sorbent towards the sorbate [21]. K_f and n determine the curvature and steepness of the isotherm [8]. The value of n also indicates the affinity of the sorbent towards the uptake of Pb^{2+}. The values for the constant and R^2 were shown on Table 3. The $1/n$ value is 0.59, and this suggests a greater sorption capacity. Also, the value of n is greater than unity for the metal ions indicating that adsorption of the metal ions was favorable.

Freundlich adsorption is commonly used to describe the adsorption characteristics for the heterogeneous surface [30]. These data often fit the empirical equation proposed by Freundlich as follows:

$$Q_e = K_f C_e^{1/n}, \quad (9)$$

where K_f = Freundlich isotherm constant (mg/g) (dm^3/g)^n and n = adsorption intensity, C_e = the equilibrium concentration of adsorbate (mg/L), Q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g). Taking logs and rearranging as follows:

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e. \quad (10)$$

The constant K_f is an approximate indicator of adsorption capacity, while $1/n$ is a function of the strength of adsorption in the adsorption process. These constants can be obtained by appropriate plot of LogQ_e against LogC_e. The constant K_f can be obtained from the intercept while $1/n$ is obtained from the slope [31]. If $n = 1$, then the partition between the two phases is independent of the concentration. If the value of $1/n$ is below unity, it indicates a normal adsorption. On the other hand, $1/n$ being above unity indicates cooperative adsorption [32]. As the temperature increases, the constants K_f and n change to reflect the empirical observation that the quantity adsorbed rises more slowly, and higher pressures are required to saturate the surface. K_f and n are parameters characteristic of the sorbent-sorbate system, which must be determined by data fitting. When performing data fitting, linear regression is generally used to determine the best isotherm models for this study [33]. $1/n$ is an heterogeneity parameter; the smaller the value of $1/n$ is, the greater the expected heterogeneity becomes. If n lies between unity and ten, this indicates a favorable sorption process [34]. From the data in Table 3 below, the value of $n = 1.02$ indicating that the sorption of Pb^{2+} unto PRH is favourable.

Temkin isotherm model was also chosen for the study of this equilibrium sorption. This was done by plotting the quantity sorbed q_e against $\ln C_e$, and the constants A_T and B were determined from the slope and intercept. These constants correlate to the adsorption capacity and intensity

Table 2: Parameters for plotting Langmuir, Freundlich, and Temkin adsorption isotherms of Pb(II) ion unto PRH.

S/N	C_o (mg/L)	C_e (mg/L)	LogC_e	Ln C_e	Q_e (mg/g)	LogQ_e	C_e/Q_e (g/L)	% Sorption
1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	10.00	0.00	0.00	0.00	10.00	1.00	0.00	100
3	50.00	0.00	0.00	0.00	50.00	1.70	0.00	100
4	100.00	6.70	0.826	1.902	93.30	1.970	0.072	93.30
5	150.00	26.94	1.430	3.293	123.06	2.09	0.22	82.04
6	200.00	62.03	1.793	4.128	137.97	2.140	0.45	68.99

Table 3: Langmuir, Freundlich, and Temkin constants for the adsorption of Pb(II) unto PRH.

Metal	Langmuir				Freundlich				Temkin			
Pb^{2+}	q_e (mg/g)	K_L (L/mg)	R_L	R^2	$1/n$	n	K_f (mg/g) (dm^3/g)^n	R^2	A_T L/mg	b_T	B	R^2
	138.89	0.699	0.0141	0.99	0.59	1.02	3.88	0.80	3.38	92.14	26.89	0.916

of adsorption. The model is given by the following equation [35]:

$$q_e = \frac{RT}{b} \ln\left(A_T C_e\right),$$
$$q_e = \frac{RT}{b_T} \ln A_T + \left(\frac{RT}{b}\right) \ln C_e, \quad (11)$$
$$B = \frac{RT}{b_T},$$
$$q_e = B \ln A_T + B \ln C_e,$$

where

A_T = Temkin isotherm equilibrium binding constant (L/g),

b_T = Temkin isotherm constant,

R = universal gas constant (8.314J/moL/K),

T = Temperature at 298 K.

Table 2 shows the parameter for plotting Langmuir, Freundlich, and Temkin adsorption isotherms, while Table 3 shows various parameters relevant to each adsorption model in the sorption of Pb(II) ion unto PRH. The Langmuir, Freundlich and Temkin adsorption isotherms are illustrated in Figures 1, 2, and 3. Considering the R^2 values for Langmuir, Freundlich, and Temkin adsorption isotherms which are 0.99, 0.797, and 0.916, respectively, examination of these plots from Figures 1, 2, and 3 suggests that the Langmuir, Freundlich, and Temkin isotherms fit the experiment, but Langmuir isotherm is a better model than Freundlich, and Temkin isotherms with respect to their R^2 values which is greater than 0.9000 and less than 1. This is an indication that PRH performed better in the sorption of Pb^{2+}, and obviously the data fitted well into the three adsorption isotherms. However, among the three isotherms, Langmuir isotherm fitted best, and this is in agreement with investigation carried out by Sumar and coworkers [36], Adekola and Adekoge [13], Adediran and coresearchers [37].

4. Conclusion

For the past few years, there is an increasing interest in the preparation of low-cost adsorbent as an alternative to biosorption of lead(II) ions. In this research, rice husk has shown its potential to be an active bioabsorbent material in solving waste water pollution as a cost-effective adsorbent. The usage of the rice husk might help to overcome part of the excessive agricultural wastes in some part of the world. This research proved that rice husks possess different physical characteristics. Chemical modification using phosphoric acid improves the adsorption capacity of active binding sites. Hence, modified rice husk is a potent and low-cost alternative adsorbent for the treatment of lead-polluted waste water.

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